भारतीय मानक
Indian Standard

IS 16918 : 2018

क्रोम-मैग्नीसाईट एवं मैग्नीसाईट-क्रोम रिफेक्ट्रीज के रासायनिक विश्लेषण के लिए पद्धति

Methods for Chemical Analysis of Chrome-Magnesite and Magnesite-Chrome Refractories

ICS 81.080

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Price Group 2

Refractories Sectional Committee, MTD 15

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Refractories Sectional Committee had been approved by the Metallurgical Engineering Division Council.

This standard is intended to be used for the assessment of the oxides of silicon, iron, aluminium, magnesium, calcium and chromium in chrome-magnesite and magnesite-chrome refractories.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

METHODS FOR CHEMICAL ANALYSIS OF CHROME-MAGNESITE AND MAGNESITE-CHROME REFRATORIES

1 SCOPE

1.1 This standard prescribes the methods for determination of oxides of silicon, iron, aluminium, magnesium, calcium and chromium in chrome-magnesite and magnesite-chrome refractories.

2 REFERENCES

The standards listed below contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and, parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of these standards.

IS No.	*Title*
460 (Part 3) : 1985	Specification for test sieves: Part 3 Methods of examination of apertures of test sieves (*third revision*)
1070 : 1992	Reagent grade water (*third revision*)
1528 (Part 7) : 2010	Methods of sampling and physical tests for refractory materials: Part 7 Methods of sampling and criteria for conformity (*second revision*)

3 SAMPLING AND PREPARATION OF SAMPLE FOR ANALYSIS

3.1 The material shall be sampled in accordance with the procedure laid down in IS 1528 (Part 7) : 2010.

3.2 The sample thus obtained shall be crushed in any type of crusher with hardened tool steel faces with due precautions to prevent contamination of the sample with steel particles from the sampling equipment during crushing and grinding. The crushed sample shall then be made to pass IS 850 µm sieve, mixed and quartered to about 20 gm. A magnet shall be gone through this 20 gm sample to remove extraneous iron particles. Thereafter, the sample shall be ground in a Tungsten carbide coated grinding media, so that it passes completely through IS 0.09 mm sieve IS 460 (Part 1) : 1985.

3.3 Sufficient quantity of the materials thus obtained shall be dried at 105-110°C for about one hour for the purpose of chemical analysis.

4 REAGENTS

4.1 Unless specified otherwise, pure chemicals and Reagent Grade Water, Grade 1 or Grade 2, IS 1070 : 1992 shall be used.

NOTE — 'Pure Chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

4.2 List of Reagents

4.2.1 *Potassium Bisulphate/Pyrosulphate*

4.2.2 *Sodium Hydroxide Solution (N/10)*

Dissolve 4 g sodium hydroxide in 1 000 ml of water.

4.2.3 *E.D.T. A Solution (M/100)*

Dissolve 3.72 g disodium salt of ethylenediamine tetracetic acid, dehydrate ($C_{10}H_{14}N_2Na_2O_82H_2O$) in distilled water, transfer to a 1 000 ml volumetric flask and dilute to the mark, mixing thoroughly.

4.2.4 *Potassium Dichromate (N/10)*

Dissolve 4.904 gm of potassium dichromate salt, previously dried at about 110 ± 10^{o}C, in water in a 1 000 ml volumetric flask and dilute to the mark. Mix the solution thoroughly.

4.2.5 *Ferrous Ammonium Sulphate (N/10)*

Dissolve 40 g salt of ammonium iron sulfate hexahydrate $\{(NH_4)_2Fe(SO_4)_2.6H_2O\}$ in water in a 1 000 ml of N/10 sulphuric acid solution in a volumetric flask. Standardized the solution against (N/10) potassium dichromate ($K_2Cr_2O_7$) solution every time it is used.

4.2.6 *Triethanolamine Solution — 30 percent (v/v).*

Dilute 60 ml of triethanolamine to 200 ml.

4.2.7 *Ammonia-Ammonium Chloride Buffer Solution (pH = 10)*

Dissolve 57.5 g of ammonium chloride in 430 ml of water and 570 ml of ammonia (sp.gr. = 0.88) and mix thoroughly.

5 DETERMINATION OF SILICA BY THE GRAVIMETRIC METHOD

5.1 Procedure A (Pyrosulphate Method)

5.1.1 Fuse 0.5 gm test sample with approximately 10 gm of potassium pyrosulfate ($K_2S_2O_7$) or sodium pyrosulfate ($Na_2S_2O_7$) in a platinum crucible over Bunsen burner or in a muffle furnace. Raise the temperature gradually to dull red heat. Most of the refractory materials should be thoroughly fused in 1 to 1.5 hours. Remove the crucible from furnace and allow it to cool in a desiccator. The pyrosulphate melt will solidify, shrink and detach from the surface of the platinum crucible, and the main mass may be removed easily in one cake.

5.1.2 Dissolve the cake and the adhering portions retained on the crucible walls in 200 ml of hot hydrochloric acid (HCl) (1:1), boiling the solution acquires a clear, bright green colour. Evaporate the dryness and bake at temperature not over 120°C until free from hydrochloric acid (HCl) fumes. Cool and moisten with hot (1:1) hydrochloric acid (HCl), then add 25 ml more acid. Warm until all salts are in solution and filter off silicon dioxide (SiO_2) (using Whatman 40 or equivalent) washing thoroughly with hydrochloric acid (HCl) (N/10) followed by washing with hot water until chloride free.

5.1.3 Again evaporate the above filtrate to dryness, bake and filter again, using Whatman 40 or equivalent. Wash the silicon dioxide (SiO_2), thoroughly with hydrochloric acid (HCl) (N/10) until chloride free and preserve the filtrate. Ignite both the precipitates of **5.1.1** and **5.1.2** in a platinum crucible to constant weight (W1). Add two to three drops of sulphuric acid (H_2SO_4) (1:1) and about 10 ml of hydrogen fluoride (HF) to the crucible. Evaporate ignite and weight (W2).

The difference of W1 and W2 represents the amount of Silicon dioxide (SiO_2) present in the sample. Fuse the residue left in crucible with pyrosulphate and add to the main filtrate, which we call Filtrate A.

5.1.4 *Calculation*

$$\% \ SiO_2 = \frac{(W1 - W2)}{W} \times 100$$

where,

W1 = mass of dish with residue, in gram;

W2 = mass of dish after hydrofluorization, in gram; and

W = weight of sample, in gram.

5.2 Procedure B (Perchloric — Sulphuric Acid Method)

5.2.1 Take 0.5 gm test sample in 400 ml beaker and add about 20 ml each of perchloric acid and 1:1 sulphuric acid (H_2SO_4). Cover the beaker and digest on hot plate. Stir the content gently and occasionally. Repeat the process two or three times, if required, until the entire sample is dissolved. Cool and dilute to 200 ml with distilled water and filter (using Whatman 40 or equivalent) with proper washing and preserve the filtrate. Ignite the residue in platinum crucible and weight (W1). Add two or three drops of 1:1 sulphuric acid (H_2SO_4) and about 10 ml Hydrogen fluoride (HF) to the crucible. Evaporate, ignite and weigh (W2). The difference of W1 and W2 represents the amount of Silicon dioxide (SiO_2) present in the sample. Fuse the residue left in crucible with pyrosulphate and add to the main filtrate, which we call Filtrate A.

5.2.2 *Calculation*

$$\% \ SiO_2 = \frac{(W1 - W2) \times 100}{W}$$

where,

W1 = mass of crucible with residue, in gram;

W2 = mass of crucible after hydrofluorization, in gram; and

W = wseight of sample, in gram.

5.3 Out of the above two processes of silicon dioxide (SiO_2) determination, the pyrosulphate method (Procedure A) shall be followed in case of any dispute.

6 DETERMINATION OF OXIDES OF CHROMIUM, ALUMINIUM, IRON, TITANIUM, CALCIUM AND MAGNESIUM

6.1 Determination of Chromium Oxide (Cr_2O_3)

6.1.1 Make up the main filtrate (A/A*) preserved at **5.1.3** or **5.2.1** to 500 ml in a volumetric flask. Take 100 ml aliquot and add 25-30 ml sulphuric acid (H_2SO_4) (1:1) and 5 ml of concentrated nitric acid (HNO_3). Heat the solution to fuming and continue fuming for few minutes. Dilute to 200 ml with distilled water and boil. Cool and add 20-25 ml silver nitrate ($AgNO_3$) (0.5%) solution and 1 to 2 drops of potassium permanganate ($KMnO_4$) followed by 10-15 ml of ammonium persulfate ($(NH_4)_2S_2O_8$) solution. Keep at low temperature for 5-10 minutes till appearance of pink colour. Now boil for about 5 minutes and add 20 ml of sodium chloride (NaCl) solution (10 percent). Again boil the solution till the permanganate colour disappears, after which continue boiling the solution for 5-10 minutes. Cool to room temperature and add 3-5 ml of phosphoric acid (H_3PO_4).

Add a measured excess of ferrous ammonium sulphate (N/10), which will change the colour of solution from amber to bright green. Add 2-3 drops of sodium diphenyl amine sulphonate as indicator and titrate with standard (N/10) potassium dichromate ($K_2Cr_2O_7$) solution. The titration should be continued till a permanent violet is obtained.

6.1.2 *Calculation*

$$\% \text{ of } Cr_2O_3 = \frac{(T_2\text{-}T_1) \times (\text{N of } K_2Cr_2O_7) \times 0.02534}{W} \times 100$$

where,

T_1 = volume of potassium dichromate ($K_2Cr_2O_7$)

T_2 = volume of measured excess of ferrous ammonium sulphate

W = weight of sample (in gram) = 0.1 gm

6.2 Determination of Ferric Oxide (Fe_2O_3)

6.2.1 Take 250 ml aliquot in a 500 ml beaker and add 10 gm ammonium chloride (NH_4Cl) and 2 to 3 drops of methyl red indicator. Heat the solution almost to the boiling and slowly add ammonium hydroxide (NH_4OH) (1:1) until the colour of the solution has changed to yellow. Boil for about 2 minutes to remove excess ammonia and allow the precipitate to settle. Filter (using Whatman 41 or equivalent) and wash with 2 percent ammonium nitrate (NH_4NO_3) solution and then with hot water. Preserve the filtrate. Dissolve the above residue in hot hydrochloric acid (HCl) (1:1). Boil and dilute with hot water and re-precipitate with ammonium hydroxide (NH_4OH) (1:1). Filter (using Whatman 41 or equivalent) the precipitate and wash it with hot water. Combine this filtrate with the earlier preserved filtrate, which we call Filtrate B, and make up to 500 ml in a calibrated flask to be used for estimation of calcium oxide (CaO) and magnesium oxide (MgO).

6.2.2 Dissolve the precipitate in hot nitric acid (HNO_3) (1:1) and add about 1 to 2 gm of potassium chlorate ($KClO_3$). Evaporate and add more potassium chlorate ($KClO_3$), if necessary, until all chromic salt (green) have been converted to chromate (orange). Dilute the solution to 300 ml, make it just ammonical, boil to coagulate the hydroxide precipitates of aluminium and iron (chromium as chromate does not interfere now). Filter (using Whatman 41 or equivalent), wash with hot 2 percent ammonium nitrate (NH_4NO_3) solution and then with hot water. Ignite the precipitate in a tared platinum crucible. Cool and weigh as mixed oxides (M).

6.2.3 Fuse the mixed oxides with 3 to 4 gm potassium bisulphate and dissolve the melt in 100 ml of 10 percent sulphuric acid and make up to 250 ml.

6.2.4 Take suitable aliquot from 250 ml and heat to boiling and reduce the iron by adding tin chloride ($SnCl_2$) drop wise until the solution is colourless. Then add 1 drop in excess. Cool quickly in running water. Add 15 ml mercuric chloride ($HgCl_2$) and 15 ml acid mixture. Titrate with standard N/10 potassium dichromate ($K_2Cr_2O_7$) solution using sodium diphenylamine sulphonate as indicator.

6.2.5 After calculating the iron (Fe) content from the titre value, convert to ferric oxide (Fe_2O_3) through the calculation given below.

Calculation:

$$Fe_2O_3 = \% \text{ Fe} \times 1.43$$

6.3 Determination of Aluminium Oxide (Al_2O_3)

6.3.1 Aluminium oxide (Al_2O_3) is obtained by difference of the weight of mixed oxides (M) obtained in **6.2.2** and the weight of Ferric oxide (Fe_2O_3) as obtained in **6.2.5**.

6.3.2 *Calculation*

$$\% \, Al_2O_3 = \left(\frac{M}{0.25} \times 100\right) - \left(\% \, Fe_2O_3\right)$$

6.4 Determination of Calcium Oxide (CaO) (by EDTA Method):

6.4.1 Take 200 ml solution from preserved filtrate (B) into a 250 ml conical flask. Add 10 ml triethanolamine solution, 5 ml of **2 N** sodium hydroxide solution drop wise while shaking the solution, and then add 0.2 g of Patton and Reeder indicator. Titrate immediately with standard EDTA solution until the colour changes from pink to blue.

6.4.2 *Calcuation*

$$\% \text{ CaO} = \frac{A \times B}{C} \times 100$$

where,

A = volume of standard EDTA solution required for titration, in ml;

B = Calcium oxide (CaO) equivalent of the standard EDTA solution, in g/ml; and

C = mass of the sample represented by the aliquot sample solution taken, in g/ml.

6.5 Determination of Magnesium Oxide (MgO) (by EDTA Method):

6.5.1 Take 200 ml of solution from preserved filtrate (B) into a 250 ml conical flask. Add 10 ml of triethanolamine solution. Add 15 ml of ammonia-ammonia chloride buffer solution. Add Eriochrome Black T indicator and immediately titrate with the standard EDTA solution until the colour changes from wine red to blue.

6.5.2 *Calculation*

$$\% \text{ MgO} = \frac{(A - B) \times C}{D} \times 100$$

where,

A = volume of standard EDTA solution required for titration of total calcium and magnesium obtained in **5.6.1**, in ml;

B = titre value for calcium;

C = Magnesium oxide (MgO) equivalent of the standard EDTA solution, in g/ml; and

D = mass of the sample represented by the aliquot taken, in g/ml.

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